Fraunhofer Institut
Kurzzeitdynamik
Ernst-Mach-Institut

Characterization of the Material Microstructure for Reactive Material Design

2nd Quarterly Progress Report I/2008

Report I-13/08

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Characterization of the Material Microstructure for Reactive Material Design
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1 Overview

The aim envisaged in this project is the development and validation of numerical methods for the mechanical simulation of materials at grain scale. These methods shall enable predictive analysis of the dependencies of the mechanical properties □ especially the fragmentation behavior □ on the morphological and constitutive nature of a material at grain scale. Simulation capability of this kind can be effectively applied to design new materials with specially tailored mechanical properties.

Specifically, this project is motivated by the aim to design (metallic) materials, which fragment under certain dynamic loading conditions into small particles, which can chemically react with a suitable ambient medium, such as shock heated ambient air or hot detonation products. Such materials could be effectively used to devise new or improved weapons with enhanced mechanical and/or thermal effects.

The desired numerical simulation capability comprises two parts. First, a method for the numerical generation of realistic microstructures must be developed. From these microstructures computational grids are generated within a representative volume element of a material using readily available meshing methods. Second, a finite-element based solver for the time dependent continuum mechanical conservation equations must be developed, which permits a three-dimensional, time dependent simulation of the response of the representative volume element to applied external loads, including a treatment of inter-grain failure and fragmentation. Both such methods are not available on the commercial software market, neither is there any satisfactory and persistent treatment known in the scientific literature yet. Within the project existing, suitable methods will be adapted and further developed towards the required capabilities.

In addition, experimental investigations will be conducted aiming at the validation of the methods and models. These experiments will consist of standard material characterization tests, microstructural analysis and specifically selected fragmentation tests. The tests and analysis will be applied to two different base materials and a mixture of those materials. The selection of suitable materials for these validation tests is also a part of this project.

Upon completion of the methods and their validation, the application of the methods will be demonstrated within a simulation based process, where the

fragmentation characteristics of the mixture material are improved by altering mixture and morphological parameters.

The project is subdivided into the following working packages:

Task 1: Structure Generator
- 1.1 Generalization for multi-material porous microstructures
- 1.2 Optimization of meshing

Task 2: Advanced Methods for Microstructural FE Solvers
- 2.1 Constitutive models
- 2.2 Appropriate interface and element formulations
- 2.3 Code performance
- 2.4 Code verification

Task 3: Material Analysis and Validation of Numerical Methods
- 3.1 Selection and procurement of materials
- 3.2 Material testing and microstructural analysis
- 3.3 Simulation of tests, evaluations and model improvements

Task 4: Virtual Material Design Process
- 4.1 Virtual improvement of fragmentation properties
- 4.2 Fragmentation tests on improved microstructures

Reporting on the work progress will be through quarterly status reports. Full technical reports will be issued instead of quarterly status reports upon completion of tasks 1 to 4, respectively.

This is the second quarterly status report. The following chapters will describe the status and progress of work in **the tasks 2, 3.1, and 3.2**. The next status report is planned for October 2008. The first full technical report is planned for June/July 2008.

2 Material Acquisition and Preliminary Material Tests

2.1 Overview

Basic requirements for the materials that shall be used as reference materials in the project were mentioned in the last quarterly report. Due to these requirements, the use of copper (Cu) and iron (Fe) powders as base materials was proposed. In discussions with Clifford Bedford from ONR during his visit (30th and 31st of January 2008) some additional desired properties were formulated which are motivated by the desired application and by the existing knowledge on reactive materials development:

- The material should preferably be manufactured by isostatic cold pressing, if the strength is too low, a heat treatment (sintering) step at a moderate temperature may follow. The material should not be sintered under pressure.
- The tensile strength of the final material should be in the range of 35 MPa (5000 PSI).
- The porosity should be around 15 %.

Based on the requirements, the use of the following base materials was proposed by Fraunhofer IFAM Dresden, the partner selected for manufacturing:

- Fe powder with a rough particle surface, see Fig. 2.1. This powder is produced in a spraying process.
- Cu powder with dendritic particles (Fig. 2.2) produced by via electrolysis.
- Alternatively, Cu powder with spherical particles produced in a spraying process (Fig. 2.3).

The Fe powder was considered suitable for use within the project, both from the simulation and the testing point of view: The particle shape is not too complicated (this would hamper the generation of microstructures for the simulation), and the strength obtained in cold pressing is sufficient such that the material can be easily handled. The dendritic Cu powder can be easily compressed and handled as well, however, it has a microstructure which is unfeasible for reproduction on the scale selected for the simulation efforts in

this project. Therefore, it was decided to use the spherical Cu powder, although its behavior in and after cold pressing is inferior to that of the dendritic powder.

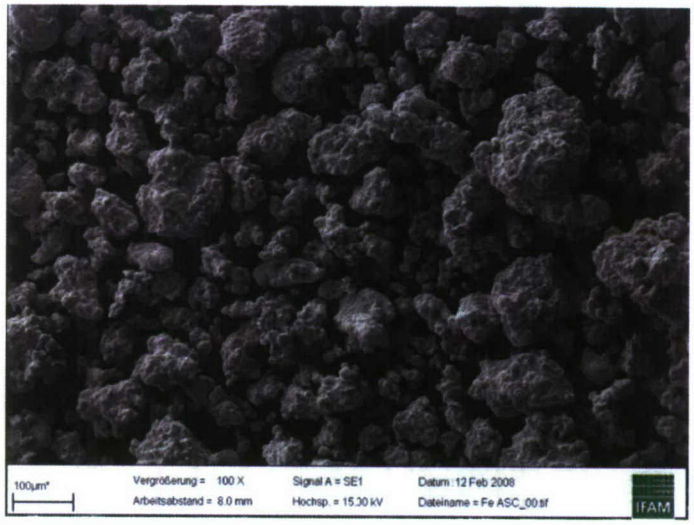

Figure 2.1: Fe powder (picture by IFAM, Dresden).

Figure 2.2: Dendritic Cu powder (picture by IFAM, Dresden, the particle in the lower right corner is an impurity).

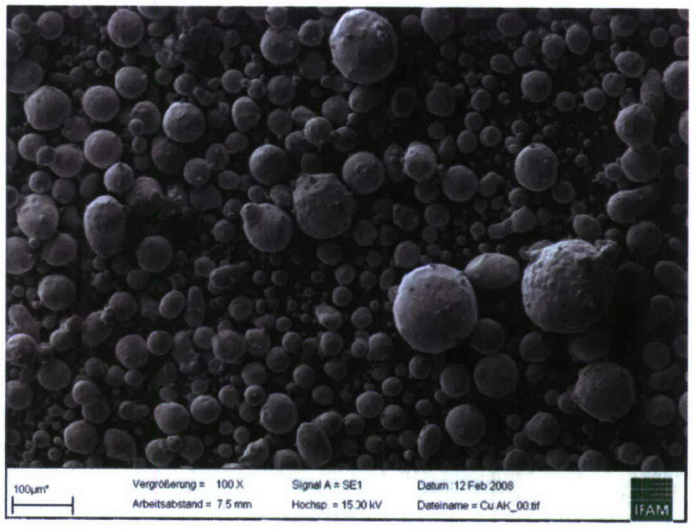

Figure 2.3: Spherical Cu powder (picture by IFAM, Dresden).

2.2 Sample preparation and testing procedure

In a first step, small discs were produced by IFAM for all three powders in order to assess the properties of cold pressed samples (Fig. 2.4). The Fe and dendritic Cu samples have a well-defined shape and sufficient strength, their porosity correlates with the pressure applied. The samples made from spherical Cu powder show some spalling on the edges and have a very low strength, they brake easily if not handled with care. Heat treatment turned out to be essential to obtain properties that are well-suited for dynamic testing of that material.

Subsequently, tensile testing specimen were produced by IFAM and tested at EMI. The specimen were fabricated using three different pressures (400, 500 and 600 MPa). Four different heat treatment schemes were applied: no heat treatment, sintered at 600 °C, 700 °C and 800 °C. The results of these tests, which are in more detail presented in the next chapters, showed that the temperature range between 700 °C and 800 °C is well suited in order to obtain the desired material properties.

Recently, the cold pressing process for spherical Cu has been extensively optimized at IFAM such that higher strength will be achieved with that material. Additional tensile testing samples will be sent to EMI until mid-April and will be tested immediately. On this basis, the sintering temperature for the first lot of dynamic testing samples can be defined. Delivery of those samples, which will be plates of 13x13 cm, is expected in May. Through the preliminary samples and testing we are now confident that both Fe and Cu materials will be appropriate for use within the project.

The samples consisted of 100 % Fe powder, 100 % Cu powder and a mixture of 50 % (by volume) of Fe powder and 50 % Cu powder.

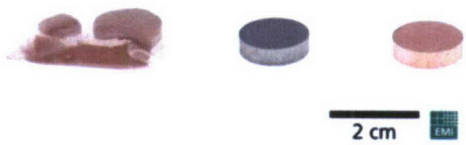

Figure 2.4: Sample material: from left to right: Cu (made from spherical powder, broken sample in plastic bag), Fe, Cu (made from dendritic powder).

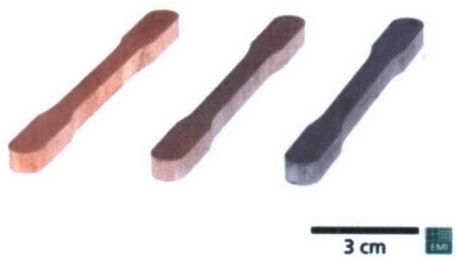

Figure 2.5: Tensile test samples: from left to right: Cu (dendritic), FeCu mixture, Fe. The geometry of the samples is according to DIN EN ISO 2740.

Optical microscopy images of the cold pressed discs are shown in Fig. 2.6-2.8. The sample made of the spherical Cu powder seems to be an unbound assembly of particles (Fig. 2.6). Bounded regions are only visible when the focus is tuned to the region between two particles. The contact area between adjacent particles is very small, so the samples are only very weakly bound. Some Cu samples were observed to break under manual handling of the samples (Fig. 2.4, left) with a strong tendency to pulverize again. It was not possible to prepare samples of the cold pressed spherical Cu powder for tensile tests.

With the dendritic Cu powder tensile test samples were successfully prepared (Fig. 2.5).

Tensile tests were executed using a tensile test machine. In all tensile tests a strain rate of $0.001 s^{-1}$ was adapted.

Experimental curves in the following relate to the machine signal of the elongation measurement.

The obtained results in most cases are from single measurements. The total error of the single measurement can be estimated to be +/- 3 %. From

measurements in which several identical prepared samples were tested, it is known that the results are fairly reproducible.

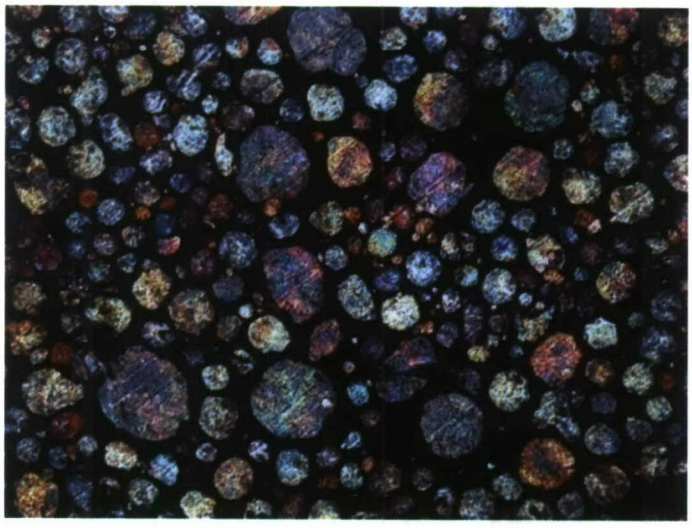

Figure 2.6: Optical microscopy image of a cold pressed Cu sample (spherical powder).

Figure 2.7: Optical microscopy image of a cold pressed Cu sample (dendritic powder).

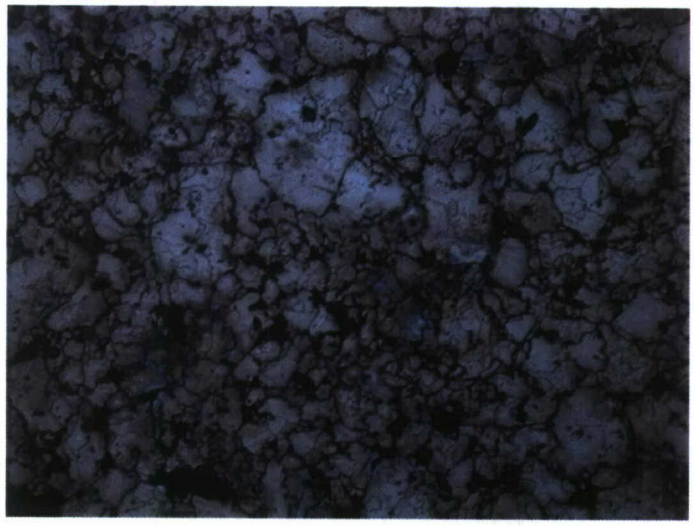

Figure 2.8: Optical microscopy image of a cold pressed Fe sample.

2.3 Samples without heat treatment

The cold pressed dendritic Cu samples reached a maximum tensile stress of 13 MPa at an elongation of 0.5 % or below (Fig. 2.9). In the case of the Fe samples, the tensile stress at break was < 4.5 MPa at an elongation < 0.5 % (Fig. 2.10). The tensile stress is lower here than in the case of the Cu (dendritic powder) sample due to a more spherical nature of the Fe powder. With increasing preparation pressure, the mechanical stability of the samples is improved.

The mechanical properties of the cold pressed spherical Cu are not sufficient for the preparation of stable samples which are suitable for the experiments.

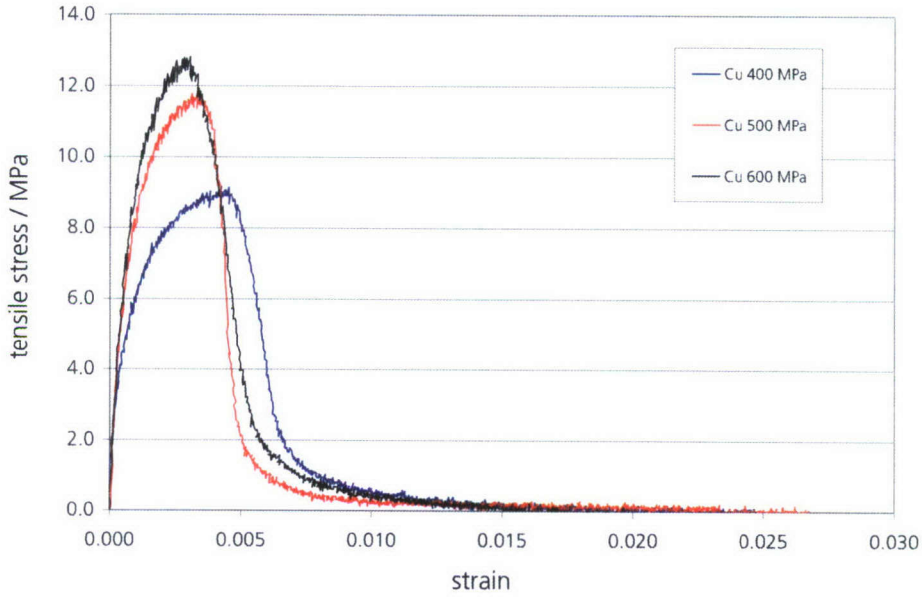

Figure 2.9: Stress-strain curves of cold pressed Cu powder (dendritic) for 3 different preparation pressures.

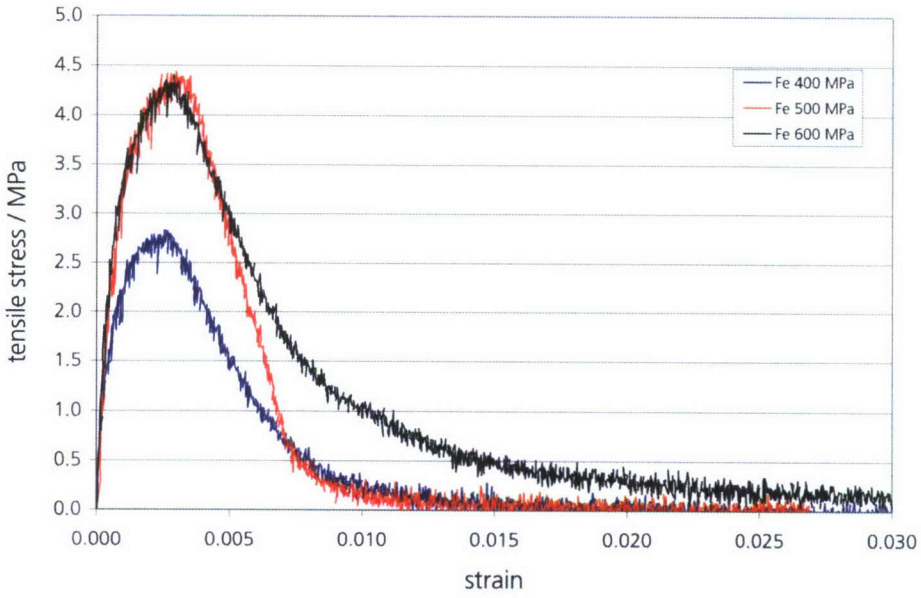

Figure 2.10: Stress-strain curves of cold pressed Fe powder for 3 different preparation pressures.

2.4 Samples with heat treatment

Further samples were prepared by IFAM from the Fe powder, the spherical Cu powder (as it is preferred from the simulation point of view) and a 50-50 mixture of these two. After a cold pressing as described above, a subsequent sintering process of 30 min duration was conducted. The sintering was executed at different temperatures of 600 °C, 700 °C and 800 °C for the investigation of the influence of the sintering temperature on the mechanical properties of the samples. Lower temperatures were not considered as, for both Fe and Cu, no sintering effects are obtained with temperatures significantly below that value.

Results of tensile tests of these cold pressed and sintered samples are discussed in the following. Measurements of the elongation were done by the built-in measurement system of the tensile test machine itself and, in addition, with a clip gauge. Note, that samples which do not break in the working region of the clip gauge can generate different values of the elongation measurement after failure initiation. The mechanical strength of all samples was sufficient for a slip free clamping in the sample holder. In almost all cases the fracture of the samples took place in between the clamping position (Fig. 2.11).

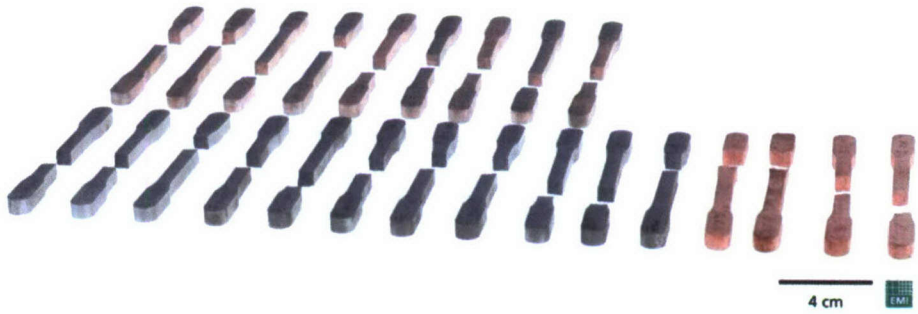

Figure 2.11: Samples after tensile test: left: Fe, right: Cu, top row: FeCu mixture.

The tensile stress at break of the Fe samples is increasing with the sintering temperature and the preparation pressure (decreasing porosity; Fig. 2.12). The ultimate elongation reaches over 7 % according to the measurement by the tensile test machine (Fig. 2.13; values at 600 °C and 800 °C fall together). The tendency in the resulting elongation values measured with a clip gauge is comparable. The Cu samples (spherical powder) are only stable enough at sintering temperatures of 700 °C or above. The resulting values for the tensile stress at break are one order of magnitude smaller than for the Fe-samples, the elongation at break is 1 % or below. The mixed samples CuFe show a maximum tensile stress between 10 and 50 MPa (Fig. 2.14) and elongations at break up to 2 % (Fig. 2.15).

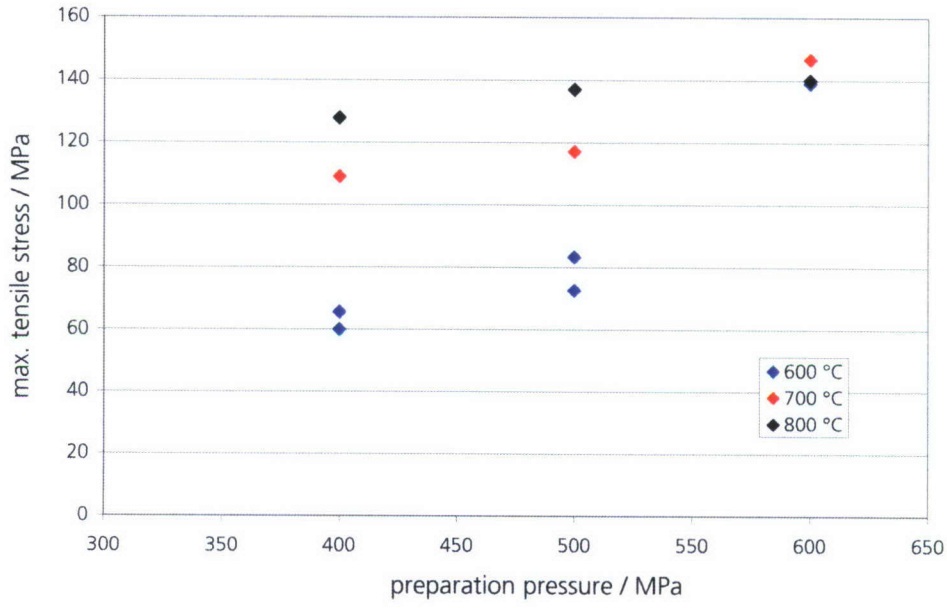

Figure 2.12: Max. tensile stress for the Fe samples with sintering temperatures of 600 °C, 700 °C, 800 °C.

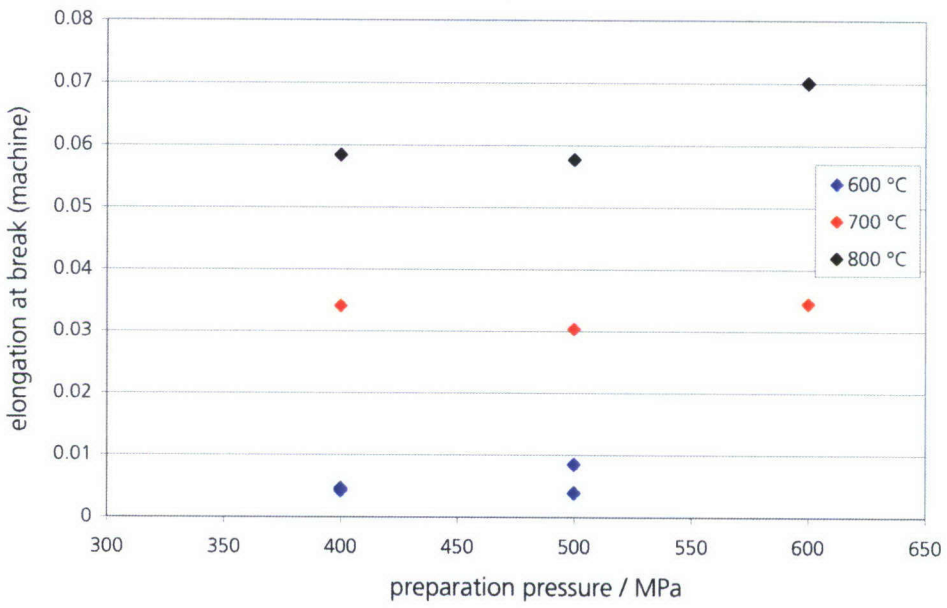

Figure 2.13: Elongation at break for Fe samples with sintering temperatures of 600 °C, 700 °C, 800 °C.

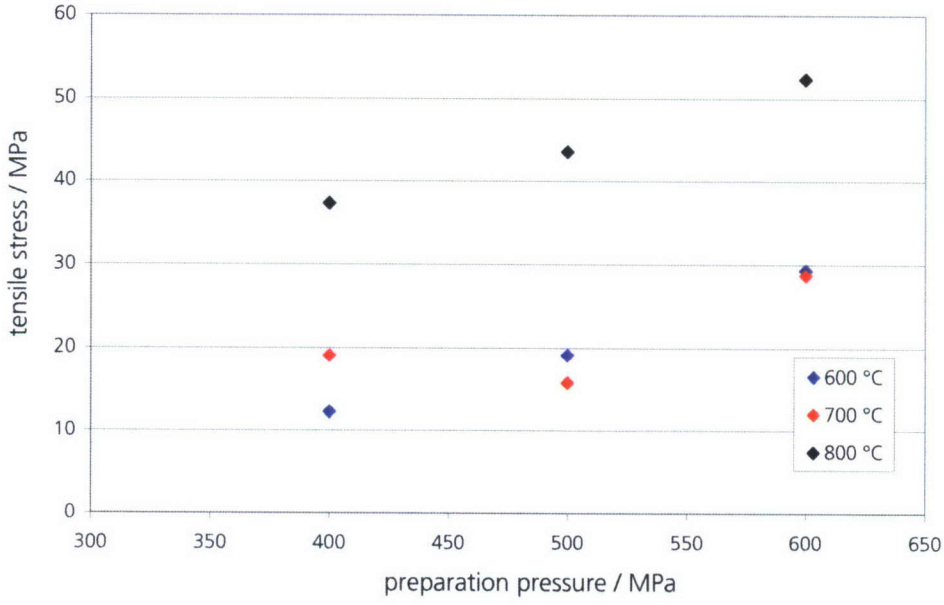

Figure 2.14: Elongation at break for FeCu samples with sintering temperatures of 600 °C, 700 °C, 800 °C.

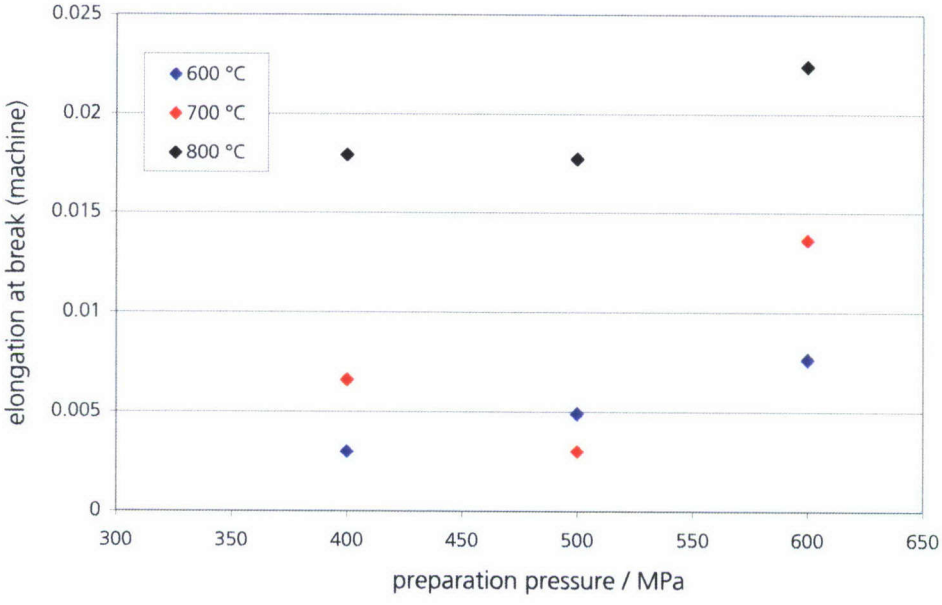

Figure 2.15: Elongation at break for FeCu samples with sintering temperatures of 600 °C, 700 °C, 800 °C.

In general the samples show a suitable dependency of the tensile strength on the sintering temperature and the preparation pressure. The tensile strength of the Fe samples is larger than that of the Cu samples. The mixed samples lie in between, what renders the possibility to adjust the mechanical properties of the mixture by relative variation of the Fe/Cu content.

2.5 Ideal sintering temperature

One parameter we have to specify for the further progress of the project is the sintering temperature. In the case of the Fe samples, 700 °C might be the optimum (Fig. 2.16), because at 800 °C the influence of the preparation pressure on the strength is diminished (Fig. 2.17). For the Cu samples, 800 °C sintering temperature seem to be more appropriate to reach a more stable material with increased tensile stress (Fig. 2.18).

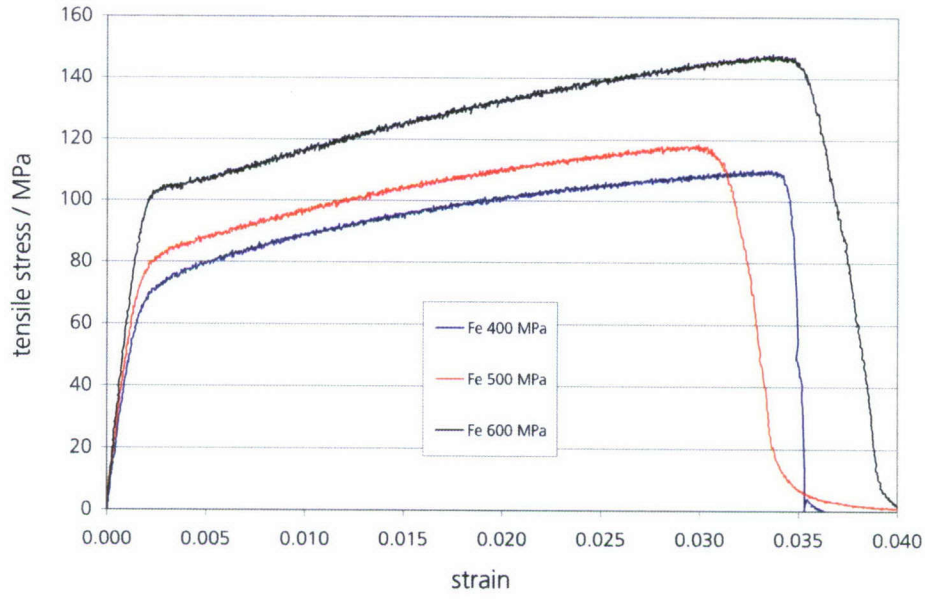

Figure 2.16: Stress-strain curves of the Fe samples at a sintering temperature of 700 °C.

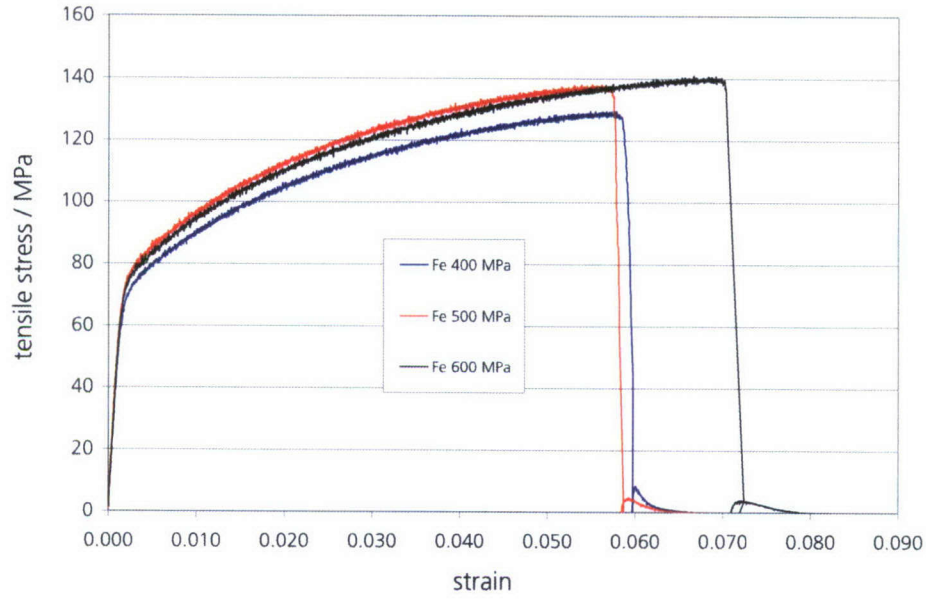

Figure 2.17: Stress-strain curves of the Fe samples at a sintering temperature of 800 °C.

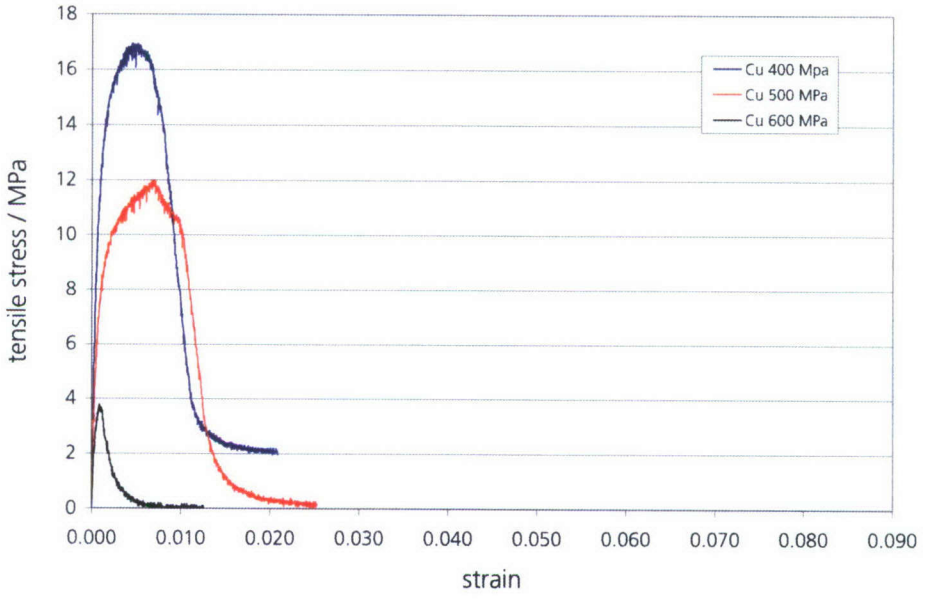

Figure 2.18: Stress-strain curves of the Cu samples at sintering temperatures of 700 °C (black curve) and 800 C (blue and red curves).

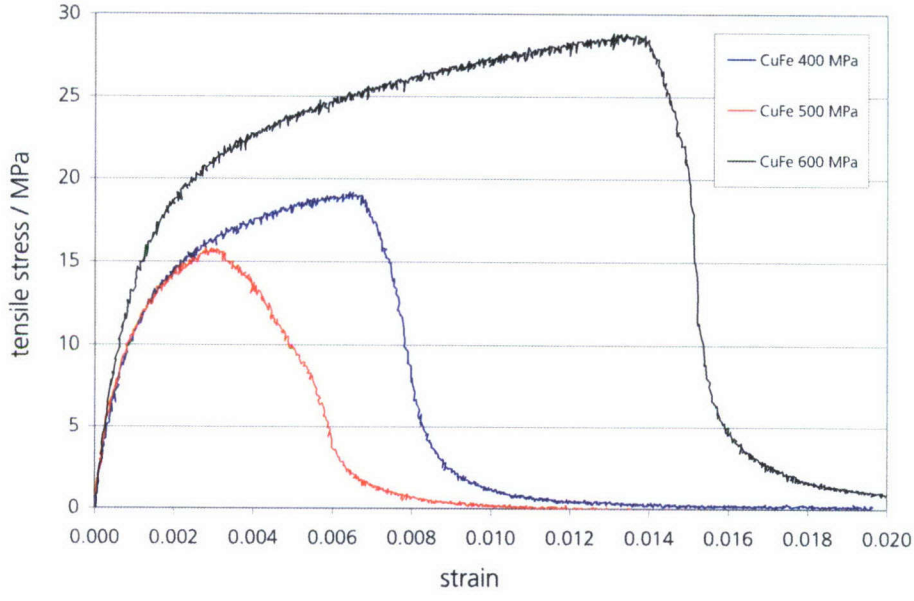

Figure 2.19: Stress-strain curves of the mixed CuFe samples at a sintering temperature of 700 °C.

All the samples tend to break very early at small elongation. Here, the sample pressed at 600 MPa delivers the smallest mechanical strength, because the break occurred nearby the clamping position of the sample. The mixed samples exhibit a material behavior which ranges between the pure Cu and pure Fe sample (Fig. 2.19). The sample prepared at 500 MPa broke very early due to a deformation caused by the preparation process and shows a smaller mechanical stability compared with the 400 MPa sample.

It would be ideal for the simulations to work with experimental data of materials which have been prepared with the same sintering temperature, so that the particle interface properties would not be influenced by this parameter. We thus plan to investigate the effect of sintering temperature in the interval between 700 °C and 800 °C in some additional experiments prior to the final specification of the sintering temperatures of the remaining lot of samples to be procured.

3 Microstructural FE Solver

3.1 Status quo

The specialized microstructural FE solver which is being developed within this project is based on an existing FE code developed at EMI. In order to represent the response of microstructures with plane grain respectively grain agglomerate interfaces, the solver has to be capable of handling simplicial triangulations. In general, this means the FE mesh has to consist of tetrahedral volume elements. The element formulation has to be able to account for metal plasticity, i.e. isochoric deformation, and has to be suited for highly transient problems. In addition, the formulation has to be efficient enough for the simulation of representative virtual material samples. These requirements limit the choice of element formulations. Based on a literature research, two alternative formulations were presented in the last quarterly report. The first alternative was a mixed tetrahedral formulation with a stabilized equal order displacement pressure interpolation proposed by Bochev et al. [2], [3] for the implicit solution of the incompressible Stokes problem. The second alternative was the F-bar method for simplicial elements proposed by de Souza Neto et al. [4].

3.2 Accomplished tasks

The examination of the element formulation proposed by Bochev et al. showed that it is not readily extended to explicit problems with non-linear material behavior and geometrical non-linearity. So the formulation was not pursued further.

The main concern regarding the F-bar method proposed by de Souza Neto was its applicability to arbitrary triangulations since the volumetric part of the deformation gradient of a simplex has to be replaced by that of a patch of at least two simplexes. Usually, in the three-dimensional case, each tetrahedron of an arbitrary primal triangulation is divided into patches of at least eight tetrahedrons to guarantee uniform and complete domain decomposition. Since each patch can be considered as a macro element, at least in regard of the pressure, an eight sub-tetrahedra element, which has eight gauss points, is inefficient compared to the composite element proposed by Thoutireddy et al. [1]. This composite element itself was not considered because of its lacking efficiency (see the last quarterly report).

A more efficient approach applicable to the F-bar method was proposed by Hauret et al. [5]. Following Hauret et al. only two simplexes are combined to a

patch and the primal triangulation is divided following a scheme that guarantees uniform and complete domain composition into two sub element macro elements. The resulting macro element is named Diamond element due to its shape.

In the following, the performance of the Diamond element, and for comparison, three other formulations is analyzed on the basis of the two-dimensional Cook's membrane which is a commonly used benchmark problem. The comparison formulations are:

1. The normal isoparametric triangle (P1P0).

2. The averaged nodal pressure triangle proposed by Burton et al. [6] (ANP). In this formulation the pressure computation is based on the dual area of the nodes which adds up pro rata from the areas of the surrounding triangles. The pressure of an element is averaged from the surrounding nodal ones.

3. The F-bar macro element composed from two adjacent triangles created by the division of a quad.

The left-hand side of the asymmetric Cook's membrane is fixed and its right-hand side exposed to a uniform vertical shear load as shown in Figure 3.1. The material of the membrane is modeled as pure elastic with a Poisson's number of 0.49999, i.e. nearly incompressible. The results of the four different formulations are displayed in Figure 3.2. All meshes shown have 162 degrees of freedom. In addition, Tab. 1 shows the maximal displacement of the loaded membranes.

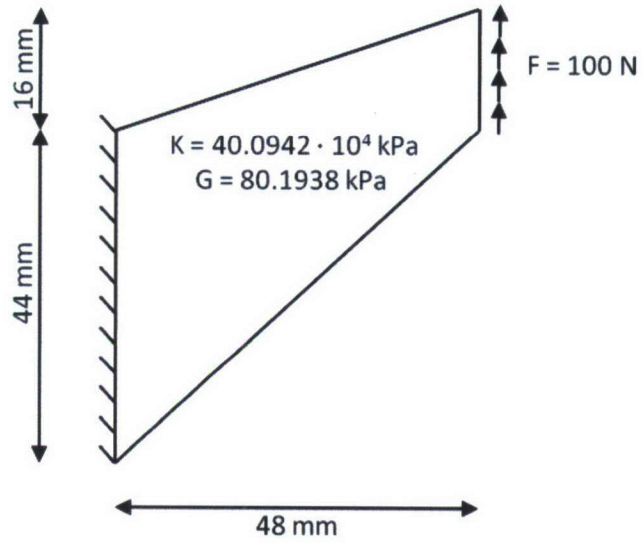

Figure 3.1: Schematic of the Cook's membrane.

As expected, the P1P0 element shows severe volumetric locking and pressure oscillations. The locking manifests in a too small tip displacement. In addition, to the pressure oscillations, the minimal and maximal pressures are significantly too low respectively too high. A correct pressure distribution is crucial for the initiation of interfacial failure. The ANP formulation overcomes the locking but shows pressure instability which is clearly identifiable by the checkerboard pressure pattern and the too low respectively too high pressure values. The F-bar and the Diamond elements exhibit comparable results without the two pathologies named above.

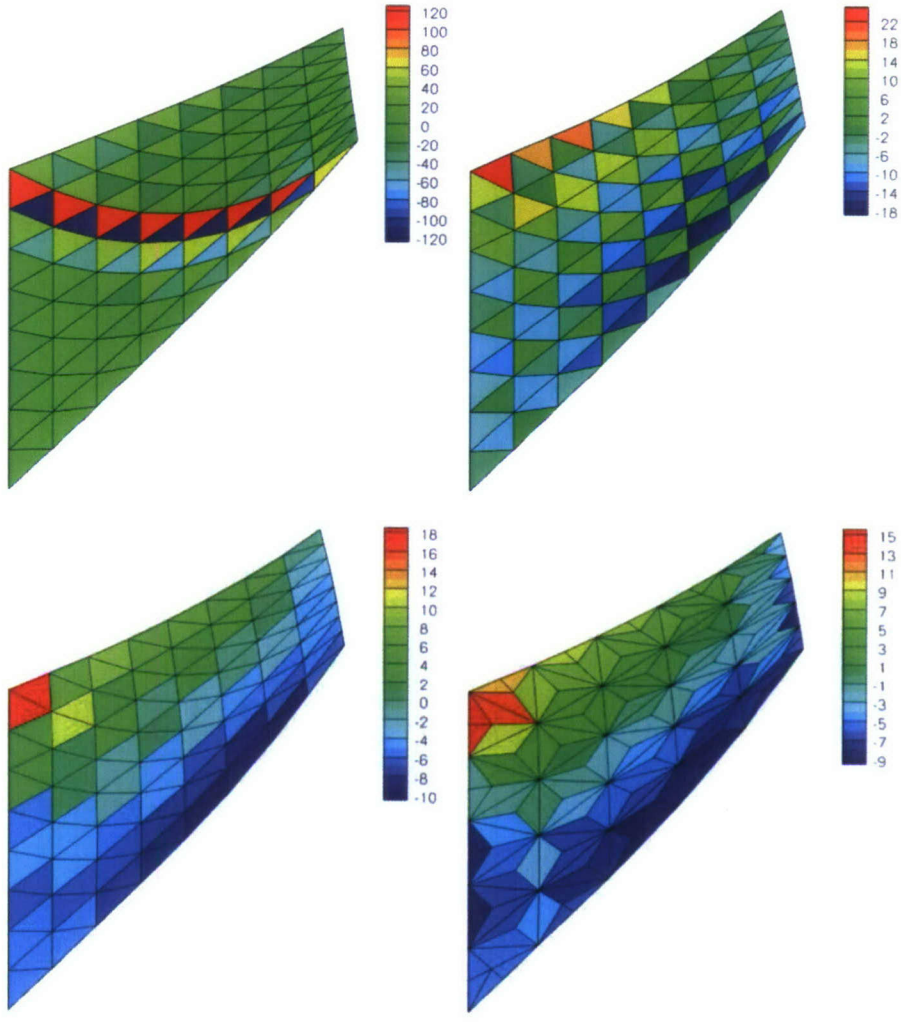

Figure 3.2: Pressure contours [kPa] of the deformed Cook's membrane. Element formulations from top left to down right: P1P0, ANP, F-Bar and Diamond.

Tab. 1: Steady state displacement [mm] of the top right tip of the deformed Cook's membrane.

	P1P0	ANP	F-bar	Diamond
Tip displacement	3.5978	6.3737	6.5100	6.3402

In addition, generic two-dimensional simulations, i.e. assuming plane strain, of a Taylor bar impact were carried out for the P1P0, ANP and F-bar element formulation. The results of these simulations confirmed the findings of the simulations of the Cook's membrane and pointed out the necessity of adopting an optimal element formulation. The results of the Taylor bar simulations will be documented in the next report.

3.3 Next steps

The F-bar method in conjunction with the special domain decomposition scheme proposed by Hauret et al. will be further analysed and evaluated. It is favoured because it shows no volumetric locking, has no pressure oscillations and is applicable to arbitrary domains. Next the formulation will be implemented in three dimensions and its compatibility to dynamically inserted initial rigid interface elements will be analysed. In addition, it will be evaluated if a different domain decomposition scheme can be developed which produces sub elements with a better aspect ratio than the one proposed by Hauret et al.

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14. ABSTRACT

The second quarterly status report addresses the current tasks "material selection and procurement", "material testing and microstructural analysis" and "microstructural Finite-element solver". A preliminary lot of candidate materials (Cu, Fe, CuFe) has been delivered and tested. The materials were manufactured by cold pressing at different pressures and subsequently sintered at different temperatures. The effects of these parameters show up clearly in the measuered values of the tensile strength and maximum strain. Based on the results of these tests the remaining lot of materials for the main series of tests will be ordered next. Concerning the microstructural FE solver, several approaches for tetrahedral elements, as found in current literature, have been tested for their applicability to explicit time integration of elastic-plastic deformations. The conducted tests showed that these schemes still suffer from locking and pressure oscillations. Further investigations on suitable element formulations are required.

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